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MANUFACTURING METHOD OF IN-MOLD COATING INJECTING MOLDING
AND INJECTING MOLDING

[Inmoeudo koteingu shashutsu seikeihin no seizo hoho and shashutsu seikeihin]

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1. A manufacturing method of in-mold coating injecting molding characterized by coating the injecting molding with polyolefin resin as the principal feed material in the dies, there are the following steps of operation:

a step of operation of primary molding in which the heated and melted feed material with polyolefin resin as the principal component is injected into a cavity formed from a male die and a female die to form a primary molding;

a step of operation of ozone contact in which the male die and the female die are separated from each other to form a space between the dies and the primary molding, and an ozone-containing gas is fed into said space to have the ozone contact the surface of the primary molding; a step of operation of gas exhausting in which the ozone-containing gas is exhausted from the dies;

and a step of operation of coating in which the coating material is injected into the space formed between the dies and the primary molding to coat the primary molding, and then the coating material is cured.

2. The manufacturing method described in Claim 1 characterized by the fact that the polyolefin resin is polypropylene resin.

3. A type of in-mold coating injecting molding manufactured using the manufacturing method described in Claim 1 or 2.

* [Numbers in right margin indicate pagination of the original text.]

Detailed explanation of the invention

[0001]

Technical field of the invention

Polyolefin resin is widely used in industry. Polyolefin resin does not contain chlorine, generates no harmful gases when incinerated, and can be recycled, so it is mainly used in automobiles and building materials as a material that is environmentally friendly.

[0002]

Prior art

Polyolefin resin is widely used in industry. Especially, as polyolefin resin does not contain chlorine, and generates no harmful gases even when incinerated, and it can be recycled, it is mainly used in automobiles and building materials as a material that is environment friendly.

[0003]

For the members made of polyolefin resin used in automobile application, the appearance is considered as important, so that usually coated members or laminates with skin material applied to them are used. However, in the recent years, in consideration of the environment problem, there is a demand on development of a type of molding made of polyolefin resin, which can omit the coating step of operation in which solvent is used in large quantity, can omit application of skin material, and have good appearance.

[0004]

Recently, as a scheme to cope with the environmental problems, people have proposed a method in which while the resin is molded, the resin molding is coated with the coating material in dies (in the following, it may be called "in-mold coating") (for example, Japanese Kokai Patent Application No. Hei 8[1996]-332655).

[0005]

However, in the conventional in-mold coating method the adherence with the in-mold coated coating material is poor, when polyolefin resin is used as the feed material of the molding because the polyolefin resin does not contain functional groups. As a result, although coating is performed, due to heating/cooling and stress in the practical application, the coating material may be separated from the polyolefin resin molding, and it may float to the top. This is undesired.

[0006]

Problems to be solved by the invention

The purpose of the present invention is to solve the aforementioned problems of the prior art by providing a manufacturing method of in-mold coating injecting molding and a type of injecting molding characterized by the fact that even for the injecting molding with functional group-free polyolefin resin as the principal feed material, it is still possible to perform injecting molding of a coating layer with excellent adherence and formation at high efficiency in the molding dies and obtain an injecting molding with good appearance.

[0007]

Means to solve the problems

The present invention pertains to the manufacturing method of in-mold coating injecting molding and a type of injecting molding with the following features.

(1) A manufacturing method of in-mold coating injecting molding characterized by the fact that in the manufacturing method of in-mold coating injecting molding by coating the injecting molding with polyolefin resin as the principal feed material in the dies, there are the following steps of operation: a step of operation of primary molding in which the heated and melted feed material with polyolefin resin as the principal component is injected into a cavity formed from a male die and a female die to form a primary molding; a step of operation of ozone contact in which the male die and the female die are separated from each other to form a space between the dies and the primary molding, and an ozone-containing gas is fed into said space to have the ozone contact the surface of the primary molding; a step of operation of gas exhausting in which the ozone-containing gas is exhausted from the dies; and a step of operation of coating in which the coating material is injected into the space formed between the dies and the primary molding to coat the primary molding, and then the coating material is cured.

(2) The manufacturing method described in (1) characterized by the fact that the polyolefin resin is polypropylene resin.

(3) A type of in-mold coating injecting molding manufactured using the manufacturing method described in (1) or (2).

[0008]

In the manufacturing method of the in-mold coating injecting molding of the present invention, a feed material with polyolefin resin as the principal component is used for forming the molding. As the

feed material, a polyolefin resin alone may be used, or a polyolefin resin composition prepared by adding other resins, rubber and additives may also be used. There is no specific restriction on the type as long as the injecting molding can be performed.

[0009]

The polyolefin resin for use in the present invention is a homopolymer or copolymer with α -olefin as the principal component. There is no specific restriction on the type of the resin, as long as it can perform injecting molding. Examples of said α -olefin include ethylene, propylene, 1-butene, 1-pentene, 3-methyl-1-pentene, 4-methyl-1-pentene, 1-hexene, 1-heptene, 1-octene, 1-decene, 1-dodecene, 1-tetradecene, 1-hexadecene, 1-octadecene, and other C2-20, or preferably C2-10, α -olefins. These α -olefins may be used either alone or as a mixture of several types.

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[0010]

Examples of the polyolefin resins that can be used in the present invention include the well-known polyethylene resin, polypropylene resin, polybutene-1, poly-4-methyl-1-pentene, ethylene-vinyl acetate copolymer, ethylene-acrylate copolymer, ethylene-methacrylate copolymer, ionomer, etc. Among them, from the viewpoint of mechanical strength and moldability, polyethylene resin and polypropylene resin are preferred, and, especially, polypropylene resin is more preferred.

[0011]

As the polyethylene resin, any of the high-density polyethylenes, middle-density polyethylenes and low-density polyethylenes may be used. As the polyethylene resin, any of the homopolymers, random copolymers and block copolymers of ethylene may be used preferably.

[0012]

Examples of the polypropylene resins that may be used include a homopolymer of propylene, and copolymers formed from propylene and C2-10 α -olefins other than propylene. In the case of copolymers, the copolymers with comonomer content of 10 mol% or less, or preferably 5 mol% or less, are preferred. Examples of the comonomers include 1-butene, 1-pentene, 3-methyl-1-pentene, 4-methyl-1-pentene, 1-hexene, 1-heptene, 1-octene, 1-dodecene, etc. Among them, ethylene is preferred. For the polypropylene resin, any homopolymer, random copolymer and block copolymer may be used preferably.

[0013]

For the polypropylene resins, they may be used either alone or as a mixture of several types. Corresponding to the application and thickness of molding, a composition of an appropriate composition selected from a homopolymer, random copolymer and block copolymer may be used in foaming molding while the desired mechanical strength is maintained and the crystallization rate is adjusted. Especially, when using a polypropylene resin with high crystallinity, it is possible to adjust the crystallization rate by adjusting the composition of the random copolymer.

[0014]

The polypropylene resin and other polyolefin resins for use in the present invention can be manufactured with the conventional method. For example, it may be manufactured by polymerization of a monomer in the presence of a catalyst system composed of solid catalyst component (a) containing magnesium, titanium, halogen and an electron donor, organic aluminum (b), and an electronic donor (c).

[0015]

As long as the purpose of the present invention is not degraded, olefin base elastomer may be added in the feed material of the molding for use in the present invention. There is no specific restriction on the type, as long as it is an amorphous elastic copolymer made of copolymer with α -olefin as the principal monomer. Examples of said α -olefin include C2-20, or preferably C2-10 α -olefins. More specifically, examples of said α -olefins include ethylene, propylene, 1-butene, 1-pentene, 3-methyl-1-pentene, 4-methyl-1-pentene, 1-hexene, 1-heptene, 1-octene, 1-decene, 1-dodecene, 1-tetradecene, 1-hexadecene, 1-octadecene, etc. These α -olefins may be used either alone or as a mixture of several types.

[0016]

Examples of the olefin base elastomers include ethylene-propylene copolymer, ethylene-butene copolymer, ethylene-hexene copolymer, ethylene-octene copolymer, ethylene-decene copolymer, and other ethylene/ α -olefin copolymers, propylene-butene copolymer, propylene-butene-ethylene copolymer, etc.

[0017]

The olefin base elastomers may be used either alone or as a mixture of several types. By adding the olefin base elastomer, it is possible to obtain a molding with excellent impact strength.

[0018]

The olefin base elastomer can be obtained using the conventional method. It can be obtained by means of copolymerization of monomers in vapor phase or liquid phase in the presence of a transitional

metal catalyst. There is no specific restriction for the catalyst and the polymerization method for manufacturing. For example, one may make use of Ziegler type catalyst, Phillips type catalyst, metallocene type catalyst, or the like in polymerization using vapor phase method, solution method, bulk polymerization method, or other polymerization method.

[0019]

As long as the purpose of the present invention is not degraded, it is possible to add an inorganic filler in the feed material of the molding for use in the present invention. There is no specific restriction on the well known inorganic filler for use in the inorganic filler, such as talc, silica, mica, calcium carbonate, glass fibers, glass beads, barium sulfate, magnesium hydroxide, wallasnite [transliteration], calcium silicate fibers, carbon fibers, magnesium oxy sulfate fibers, potassium titanate fibers, titanium oxide, calcium sulfite, white carbon, clay, calcium sulfate, etc. The inorganic fillers may be used either alone or as a mixture of several types. By adding an inorganic filler, it is possible to obtain a molding with high rigidity and high dimensional precision.

[0020]

As long as the purpose of the present invention is not degraded, it is possible to add resins other than polyolefin resin, copolymer rubber other than said olefin base elastomer, conjugate diene rubber or other rubber-like polymer, etc. appropriately in the feed material of the molding for use in the present invention. Also, as needed, it is possible to add various types of additives. Examples of the additives include nucleating agent, oxidation inhibitor, hydrochloric acid absorbent, heat resistant agent, weatherproof stabilizer, photo-stabilizer, UV absorbent, slipping agent, anti-blocking agent, fog

inhibitor, lubricant, static inhibitor, flame retarding agent, pigment, dye, dispersant, copper hazard inhibitor, neutralizer, foaming agent, plasticizer, foam inhibitor, crosslinking agent, peroxide, and other fluidity improving agent, weld strength improving agent, natural oil, synthetic oil, wax, and other well known additives.

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[0021]

Said olefin base elastomer, inorganic filler, resin, rubber-like polymer and various additives for adding in the feed material of the molding may be used either alone or as a mixture of several types in another combination.

[0022]

For the feed material of the molding for use in the present invention, that is, polyolefin resin or polyolefin resin composition, especially polypropylene resin or polyolefin resin composition, especially polypropylene resin or polypropylene resin composition, the melt flow rate (MFR) measured at 230°C and under the load of 2.16 kg according to ASTM D1238 should be in the range of 1-200 g/10 min, or preferably in the range of 2-150 g/10 min. When the MFR is within said range, not only it is possible to perform high speed injection, but also it is easy to adjust the balance between rigidity and impact strength.

[0023]

There is no specific restriction on the coating material, and the conventional coating material (paint) may be used. Examples include the one-liquid type coating materials mainly made of the binder components that can be cured by a peroxide crosslinking agent, such as polyfunctional acrylate resin,

polyfunctional methacrylate resin, unsaturated polyester resin, epoxy acrylate oligomer, urethane acrylate oligomer, etc., and two-liquid type coating materials prepared by mixing the principal agent and the curing agent right before injecting into the dies, such as epoxy resin/polyamine curing system, polyol resin/polyisocyanate curing system, etc.

[0024]

According to the present invention, the aforementioned feed material is used to manufacture injection molding by means of melt blending and then injection molding, and with coating performed in the dies. The injecting molding device and dies used in the molding operation have the following characteristics.

- 1) It is possible to change the internal volume of the cavity.
- 2) The ozone-containing gas can be fed into the cavity and can be exhausted.
- 3) It is possible to inject the coating material into the cavity.

[0025]

As the ozone-containing gas for use in the present invention, one may make use of ozone gas. However, it is preferred that an ozone-containing gas prepared by diluting the ozone gas with air, nitrogen, carbon dioxide, oxygen, or other carrier gas be used. The concentration of ozone in the ozone-containing gas should be in the range of 0.5-300 g/m³, or preferably in the range of 1-200 g/m³.

[0026]

It is possible to obtain the well known ozone generator to generate the ozone-containing gas. Examples of the ozone generating devices include (i) a device generating ozone by electric

decomposition of moisture in air; (ii) a device generating ozone by discharge treatment of oxygen in air; (iii) a device generating ozone by discharge treatment of pure oxygen.

[0027]

According to the manufacturing method of the present invention, in the primary molding operation, the heated and melted feed material of said molding is injected into a cavity to form a primary molding. The injecting molding can be performed using the same method as that of the conventional injecting molding.

[0028]

In the ozone contact operation, the male die and/or the female die are driven to move so that the male die and the female die are separated from each other. As a result, a space is formed between the dies and the primary molding. Here, the ozone-containing gas is fed into the space, so that ozone contacts the primary molding. It is also possible to perform movement of the dies (formation of space) while the ozone-containing gas is fed (injected). The contact time between ozone and the primary molding should be in the range of 1-300 sec, or preferably in the range of 10-100 sec.

[0029]

By means of said ozone contact treatment, the surface of the primary molding is oxidized by ozone to be activated. As a result, even when polyolefin resin free of functional groups is used as the feed material of molding, it is possible to improve the adhesive strength between the molding and the coating material, and it is possible to form a coating layer (coating film) with excellent adhesive strength.

[0030]

In the gas exhausting operation, by substituting with air, or evacuation, or driving the movable die, the cavity clearance can be reduced, so that the ozone-containing gas for use in the ozone contact operation is exhausted from the dies. The exhausted ozone-containing gas can be treated using the conventional treatment method.

[0031]

In the coating operation, said coating material is injected into the space formed between the dies and the primary molding, so that the coating material is uniformly spread on the surface of the primary molding for coating, followed by curing of the coating material to form a coating layer. After end of said gas exhausting operation, if the space between the dies into which the coating material is injected and the primary molding cannot be guaranteed, for example, the movable die is moved to reduce the cavity clearance to exhaust the ozone-containing gas. The dies are moved to guarantee the space needed for injecting the coating material, then the coating material is injected. In the coating operation, coating with excellent adhesive strength on the molding surface is applied to form an injecting molding. The thickness of the coating layer should be in the range of 1 μm - 3 mm, or preferably in the range of 10 μm - 1 mm, or more preferably in the range of 10 μm - 100 μm .

[0032]

For the in-mold coating injecting molding obtained in this way, there is an excellent adhesive strength between the coating layer and the polyolefin resin layer as the base material, and separation can hardly take place even in the cold/hot cycle test. Also, compared with the outer coating of dies, the inner surface of the dies can be transferred well to the coating layer. Consequently, good coating film is

formed on the mirror surface and the embossed surface, and a molding with excellent appearance can be obtained.

[0033]

The in-mold coating injecting molding of the present invention is the type obtained in the manufacturing method of the in-mold coating injecting molding.

[0034]

The in-mold coating injecting molding of the present invention can be used in various fields, such as door trims, instrument panels, and other inner parts of automobiles, side protecting mirrors, bumpers, soft facers, mud guards, and other automobile outer parts, housing for home appliances, office appliances, groceries, kitchen appliances, building materials, sports appliances, etc.

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[0035]

Effect of the invention

According to the manufacturing method of the in-mold coating injecting molding of the present invention, after contact between the ozone-containing gas and the injecting molding in the dies, a coating material is injected into the dies to form a coating layer. Consequently, for the injecting molding with functional group-free polyolefin resin as the principal feed material, it is possible to form a coating layer with excellent adhesive strength in the molding dies while injecting molding is performed, and it is possible to manufacture the injecting molding with good appearance at a high efficiency. The in-mold coating injecting molding of the present invention is of the type manufactured by said manufacturing

method. A coating layer with good adhesive strength to the injecting molding with functional group-free polyolefin resin as the principal feed material is formed, and it has a good appearance.

[0036]

Embodiment of the invention

In the following, an explanation will be given regarding an example of the manufacturing method in the present invention with reference to figures. Figure 1 illustrates the steps of operation in an example of the manufacturing method of the in-mold coating injecting molding in the present invention.

Figure 1(a) shows the state before start of injection of the polypropylene resin composition; Figure 1(b) shows the state at end of injection; Figure 1(c) shows the state at end of filling of the ozone-containing gas; Figure 1(d) shows the state at the end of exhaustion of ozone-containing gas; Figure 1(e) shows the state of injection of the coating material; and Figure 1(f) shows the state of curing of the coating material. The manufacturing process goes in the order of steps (a), (b), (c), (d), (e) and (f).

[0037]

As shown in Figure 1, (1) represents a female movable die, and (2) represents a male stationary die. They form the dies for the injecting molding. Said movable die (1) can be driven to move left/right with respect to stationary die (2). The structure is such that as movable die (1) is pressed into stationary die (2), cavity (3) is formed inside the dies. Here, the dies have the so-called shear edge structure that can prevent leak of the polypropylene resin composition filled in the dies even when the cavity volume varies.

[0038]

In movable die (1), ozone injector (4), which feeds the ozone-containing gas into cavity (3), and coating material injector (5), which injects the coating material in cavity (3), are set. Said ozone injector (4) is connected to an ozone generator set out of the dies through a pipe made of corrosion resistant metal or Teflon (registered trademark) or other ozone-resistant material, so that the ozone-containing gas can be fed to cavity (3). Here, however, the pipe and the ozone generator are not shown in the figure.

[0039]

Also, coating material injector (5) is connected via a flexible tube to a coating material ejecting pump set out of the dies, and the coating material stored in the coating material tank is injected into cavity (3) as the ejecting pump is turned on. Here, the flexible tube, coating material ejecting pump and coating material tank are not shown in the figure. (7) represents the ejecting device, (8) represents the polypropylene resin composition as the feed material of the molding, and (9) represents a feed stalk.

[0040]

When the in-mold coating injecting molding is manufactured with the device shown in Figure 1, first of all, from the state shown in Figure 1(a), movable die (1) is driven to move forward to close the dies. Then, by means of ejecting device (7), melted polypropylene resin composition (8) is injected to fill in cavity (3) from said feed stalk (9), followed by cooling and solidification to form primary molding (10) (Figure 1(b), primary molding operation).

[0041]

The resin temperature of injected polypropylene resin composition (8) should be in the range of 170-270°C, or preferably in the range of 180-260°C. Also, the pressure in the molding dies should be in the range of 5-20 MPa, or preferably in the range of 10-15 MPa.

[0042]

Then, movable die (1) is driven to move to have primary molding (10) and movable die (1) separated from each other to form secondary space (11) between primary molding (10) and movable die (1). In this secondary space (11), ozone-containing gas (12) with ozone concentration in the range of 0.5-300 g/m³, or preferably in the range of 1-200 g/m³, is injected from ozone injector (4), and it contacts primary molding (10) in ozone-containing gas (12) (Figure 1(c), ozone contact operation). Injection of ozone-containing gas (12) is performed at the same time of the movement operation of movable die (1), and, as a result, the dies opening operation can be performed smoothly. This is preferred. The injection quantity of ozone-containing gas (12) into secondary space (11) matches the volume of secondary space (11). For example, for a molding with projection area of 1 m², when the movement distance of movable die (1) is 2 mm, about 2 L can be fed.

[0043]

The time of molding between ozone-containing gas (12) and primary molding (10) should be 1-300 sec, or preferably in the range of 10-100 sec. By means of this ozone contact operation, the surface of primary molding (10) exposed to ozone-containing gas (12) is activated by oxidation reaction, so that the surface has excellent adhesive strength with the coating material to be coated in the next step of operation.

[0044]

After said contact time, movable die (1) is driven to move to contract the cavity clearance, so that ozone-containing gas (12) is exhausted through an exhausting hole (not shown in the figure) set in the shear edge portion (Figure 1(d), gas exhausting operation). Said exhausted ozone-containing gas (12) is treated using the conventional treatment method.

[0045]

Then, movable die (1) is driven to move to form secondary space (11), and coating material (13) is injected from coating material injector (5) into said secondary space (11) to form a layer with thickness in the range of $1\text{ }\mu\text{m}$ - 3 mm , or preferably $10\text{ }\mu\text{m}$ - 1 mm , or more preferably in the range of $10\text{ }\mu\text{m}$ - $100\text{ }\mu\text{m}$ (Figure 1(e), coating operation). Then, movable die (1) is driven to move forward, and the dies are fastened. After coating material (13) is spread uniformly, it is cured (Figure 1(f), coating operation). As a result, one can obtain in-mold coating injecting molding (14) with the surface of primary molding (10) coated with coating material (13).

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[0046]

More specifically, for example, with the initial molding clearance of 1.0 mm , a portion of polypropylene resin composition (8) is injected into cavity (3) to form primary molding (10), followed by retreating to have a molding clearance of 2.0 mm , while ozone-containing gas (12) is fed to secondary space (11). After setting at this state for 20 sec , movable die (1) is driven to move forward by 1.9 mm , so that ozone-containing gas (12) is exhausted. Then, as coating material (13), the thermosetting acrylic resin is fed from coating material injector (5), and movable die (1) is driven to

move forward to have the dies fastened. After coating material (13) is spread uniformly, it is set at this state for 30 sec for curing. Then, the dies are opened and the molding is taken out, obtaining in-mold coating injecting molding (14).

[0047]

Said in-mold coating injecting molding (14) prepared in the above has excellent adhesive strength with the coating layer and the polypropylene resin composition layer as the base material, and separation can hardly take place in the heating/cooling cycle test.

[0048]

Figure 1 is a diagram illustrating the case when polypropylene resin composition (8) is used as the feed material of the molding. However, other feed materials may be used in place of polypropylene resin composition (8). Figure 1 shows one ozone injector (4) and one coating material injector (5). However, one may make use of two of each of them. Also, the positions for setting have to be changed at will. Said ozone injector (4) has a structure that also can exhaust the gas. In this case, ozone-containing gas (12) can be exhausted from ozone injector (4). Also, as shown in Figure 1, by moving movable die (1) to close the dies, ozone-containing gas (12) is exhausted from secondary space (11). While ozone-containing gas (12) is kept, air is used to purge it, or it may be evacuated.

[0049]

Application examples

Application Examples 1, 2 and Comparative Example 1

For the in-mold coating injecting molding prepared using the method shown in Figure 1, the adhesive strength between the coating layer and the polypropylene resin composition was tested. Also, as the feed material of the molding, a polypropylene resin composition consisting of 90 parts by weight of polypropylene resin and 10 parts by weight of talc was used. As the coating material, a curing type acrylic resin paint containing a polyfunctional acrylic resin mixture and a peroxide was used. The molding condition and results are listed in Table I.

[0050]

TABLE I

	Application Example 1	Application Example 2	Comparative Example 1
Ozone concentration (g/m ³)	5	0.5	0
Adhesive strength of coating film (number/number) *1	100/100	66/100	0/100

*1. Adhesive strength of coating film: On the coating surface, 100 checkerboard squares each measuring 25 mm square are formed by ruling 11 lines in each of the longitudinal and lateral directions. Then, an adhesive tape is applied to it and is then peeled off. Then, the number of the squares left on the coating film is counted.

Brief description of the figures

Figure 1 illustrates the steps of operation in an example of the manufacturing method of the in-mold coating injecting molding in the present invention.

(a) shows the state before start of injection of the polypropylene resin composition

(b) shows the state after end of injection

(c) shows the state after end of filling of the ozone-containing gas

(d) shows the state after end of exhausting of the ozone-containing gas

(e) shows the state of injection of the coating material

(f) shows the state of curing of the coating material

Explanation of symbols

- | | |
|----|---------------------------------|
| 1 | Movable die |
| 2 | Stationary die |
| 3 | Cavity |
| 4 | Ozone injector |
| 5 | Coating material injector |
| 7 | Ejecting device |
| 8 | Polypropylene resin composition |
| 9 | Feed stalk |
| 10 | Primary molding |
| 11 | Secondary space |
| 12 | Ozone-containing gas |

13 Coating material

14 In-mold coating injecting molding

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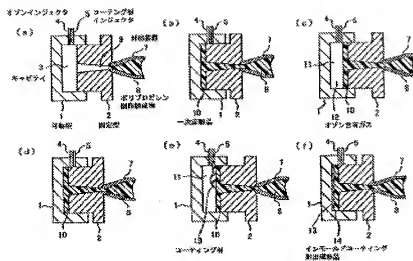


Figure 1

- Key:
- 1 Movable die
 - 2 Stationary die
 - 3 Cavity
 - 4 Ozone injector
 - 5 Coating material injector
 - 7 Ejecting device
 - 8 Polypropylene resin composition
 - 10 Primary molding
 - 12 Ozone-containing gas
 - 13 Coating material
 - 14 In-mold coating injecting molding